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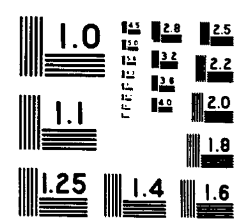

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COLUMBIUM AND TANTALUM ALLOY DEVELOPMENT

By Herbert R. Babitzke, Laurance L. Oden, and Hal J. Kelly

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COLUMBIUM AND TANTALUM ALLOY DEVELOPMENT

by

Herbert R. Babitzke,[1] Laurance L. Oden,[1] and Hal J. Kelly[2]

ABSTRACT

As part of a project to develop refractory metal alloys suitable for high-temperature structural applications, the Bureau of Mines applied solid solution and precipitation-hardening techniques to columbium and tantalum alloys. Thirty-three alloys were evaluated to determine their formability, strength, and oxidation resistance. —> p. 10

Three alloys had tensile strengths near 40,000 psi at 1,200° C: Cb-1N-5W-5V-10Hf (No. 4), Cb-15Hf-5W-0.5B (No. 31), and Cb-15Hf-5W-1B (No. 32). Oxidation resistance of the high-strength alloys was good. Alloy 4 gained only 34 mg/cm^2 at 1,200° C, and 21 mg/cm^2 at 1,000° C in 2 hours. Hot forming was done without any protection from oxidation.

INTRODUCTION

Alloying research by industry and the Bureau of Mines has improved the oxidation resistance of columbium and increased its high-temperature strength. Columbium-base alloys rank among the most promising materials for use at temperatures of 1,100° to 1,370° C with high strength-to-weight ratios. Research and development programs on alloys for high-temperature service have favored columbium over tantalum because of its lower density and lower neutron cross section and because resources of columbium-bearing minerals are more extensive than those of tantalum. Although there are indications that tantalum alloys are useful above 1,300° C, long life at these high temperatures will depend on the development of suitable oxidation-resistant coatings. For all these reasons, more emphasis has been placed on research with columbium than with tantalum.

Much research has been conducted for improving columbium and tantalum alloys over the past 20 years. Some of the alloys developed have found industrial application; however, none have the desired combination of strength,

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oxidation resistance, and formability necessary for high-temperature applications. The strength of columbium and tantalum has been improved by alloying for both solid solution strengthening and precipitation hardening, but many of the resulting alloys cannot be fabricated. For example, alloy F-48 (Cb-15W-5Mo-1Zr, or in atomic percent, Cb-8W-5Mo-1Zr), is one of the strongest alloys but has poor oxidation resistance and is difficult to fabricate. Other alloys have high strength and good formability, but for use at high temperatures the oxidation resistance is far from adequate.

This Bureau of Mines investigation was conducted to determine the effect of several alloying additions on the oxidation resistance and the strength of columbium and tantalum at high temperatures. The results expand data previously collected and published by the Bureau of Mines (1-7, 18, 21)[3] and by such firms as Battelle Memorial Institute (8-10, 13-17), Wright Air Development Center (11), Westinghouse Electric Corp. (12), and Wah Chang Corp. (19-20). Alloys developed at the Bureau of Mines during this investigation compare favorably with those made previously and are equivalent in strength to those developed by previous investigators.

PREPARATION OF ALLOYS

Experimental alloys were prepared by adding minor elements to columbium and tantalum. Analyses of columbium and tantalum, purchased as electron-beam-melted stock, are shown in table 1. The alloys were prepared first as three 50-gram buttons by arc-melting in a helium atmosphere furnace with a nonconsumable, tungsten electrode. These small buttons were melted three times and then consolidated into one larger button, 1 inch wide by 3 inches long, by melting twice more. Microscopic examination of sections cut from the buttons revealed no massive segregation.

TABLE 1. - Impurity analyses of columbium and tantalum, ppm

Element	Columbium	Tantalum	Element	Columbium	Tantalum
Aluminum.....	<20	17 - 20	Manganese.....	<20	<10
Boron........	<1	<1	Molybdenum....	<20	<10
Cadmium......	<5	<1	Nickel........	<20	<10
Carbon.......	<20 - 80	<30	Nitrogen......	25- 30	20- 25
Chromium.....	<20	<10	Oxygen........	60- 70	<50
Cobalt.......	<10	<5	Silicon.......	<50	<10
Columbium....	-	<50	Tantalum......	<500	-
Copper.......	<40	<2 - 2	Tin...........	<10	<10
Hafnium......	<80	(1)	Titanium......	<40	<10
Hydrogen.....	2.1- 2.3	1.7- 2.0	Tungsten......	250- 270	<10
Iron.........	<50	<15	Vanadium......	<20	<10
Lead.........	<20	<5	Zinc..........	(1)	<10
Magnesium....	<20	<10	Zirconium.....	<250	<50

[1]No analysis.

[3]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

To add certain elements to the columbium alloys, master alloys were prepared with the following compositions: columbium with 30 weight-percent tungsten, columbium with 4 weight-percent boron, columbium with 14.3 weight-percent carbon, and columbium with 2.4 and 2.9 weight-percent nitrogen. Adding tungsten, boron, carbon, and nitrogen in this way resulted in less segregation than when small amounts of these elements were added directly to the columbium. For the tantalum alloys all the elements were added directly, as were the other additions to columbium.

The alloys prepared are shown in table 2. To facilitate discussion, the alloys are grouped as follows: Group 1 alloys were prepared to determine the effects of aluminum, carbon, chromium, copper, hafnium, iron, nitrogen, silicon, titanium, tungsten, vanadium, and zirconium on the strength and oxidation resistance of columbium. Group 2 comprises alloys in which zirconium was substituted for the hafnium in the Cb-15Hf-5W alloy to compare results with the two elements. Group 3 alloys are based upon Cb-15Hf-5W with additions of carbon, nitrogen, and boron. Group 4 comprises tantalum alloys. The columbium-iron alloy button of group 1 shattered during cooling, and was consequently dropped from the investigation. Group 3 alloys were prepared in duplicate.

TABLE 2. - Alloys prepared for this investigation[1]

(Alloy composition in atomic percent)

Alloy	Composition	Alloy	Composition
Group 1:		Group 3:	
1........	Cb-1N-5W-3V-5Hf	13a[2]......	Cb-15Hf-5W
2........	Cb-1N-5W-5V-5Hf A	24........	Cb-15Hf-5W-0.1C
3........	Cb-1N-5W-3V-10Hf	25........	Cb-15Hf-5W-0.5C
4........	Cb-1N-5W-5V-10Hf	26........	Cb-15Hf-5W-1.0C
5........	Cb-1N-5W-3Si-5Hf B	27........	Cb-15Hf-5W-0.1N
6........	Cb-1N-5W-3Cr-5Hf C	28........	Cb-15Hf-5W-0.5N
7........	Cb-15Hf-5W-4Si D	29........	Cb-15Hf-5W-1.0N
8........	Cb-15Hf-5W-4Si-1N E	30........	Cb-15Hf-5W-0.1B
9........	Cb-15Hf-5W-2Si-2Al F Cb	31........	Cb-15Hf-5W-0.5B
10........	Cb-15Hf-5W-2Zr-4Al-1C	32........	Cb-15Hf-5W-1.0B
11........	Cb-15Hf-5W-2Zr-1C		
12........	Cb-15Hf-2Zr-4V-1C		
13........	Cb-10Ti-4Al-0.1Cu-2W		
14........	Cb-14.6Ti-3.9Zr-13.3Hf-3.5Al-0.7N K		
15........	Cb-14.8Ti-3.9Zr-13.4Hf-3.4Si L		
18........	Cb-69Fe M Cb		
Group 2:		Group 4:	Ta Ta
19........	Cb-5Zr-5W	16.......	Ta-5W-5Mo S
20........	Cb-10Zr-5W	17.......	Ta-15Hf-5W-2Re T
21........	Cb-15Zr-5W		Ta Ta
22........	Cb-20Zr-5W M		
23........	Cb-33Zr-5W		

[1]Nominal values are used throughout the paper.
[2]Data from sample 13a correspond to a sample in reference 6, p. 22.

EVALUATION OF ALLOYS

Fabrication

Sections from all of the buttons were hammer-forged at room temperature to achieve a 50-percent reduction in overall thickness. For those alloys that cracked upon cold forging, another section from the same button was hammer-forged at 1,200° C. The information obtained was used as a guide in forging the remaining portion of the button. A smaller reduction was made for the hot-forged specimens than for the cold-forged specimens because it was necessary to grind the hot-forged specimens to remove surface contamination.

Table 3 shows that the fabricability of group 1 alloys was decreased most by the silicon additions. However, the alloys containing silicon and no tungsten could be hot-forged. Hot forging was also required for the columbium-zirconium-tungsten alloys in group 2, whereas all the alloys in group 3 could be cold-forged. Compared with the results of the Cb-Hf-W alloys studied in a previous investigation (4), the rhenium addition to the tantalum alloy in group 4 reduced ductility.

TABLE 3. - Columbium and tantalum alloy hammer forging results

Alloy	Alloy composition, atomic percent	Reduction, percent		Results[1] [2]			
		Room temp.	1,200° C	Poor	Fair	Good	Excellent
Group 1:							
1.....	Cb-1N-5W-3V-5Hf..................	47	31	x	-	-	y
2.....	Cb-1N-5W-5V-5Hf..................	49	38	x	-	y	-
3.....	Cb-1N-5W-3V-10Hf.................	52	36	x	-	-	y
4.....	Cb-1N-5W-5V-10Hf.................	46	27	x	-	-	y
5.....	Cb-1N-5W-3Si-5Hf.................	45	46	xy	-	-	-
6.....	Cb-1N-5W-3Cr-5Hf.................	53	31	x	-	-	y
7.....	Cb-15Hf-5W-4Si...................	50	([3])	xy	-	-	-
8.....	Cb-15Hf-5W-4Si-1N................	50	([3])	xy	-	-	-
9.....	Cb-15Hf-5W-2Si-2Al...............	42	([3])	xy	-	-	-
10.....	Cb-15Hf-5W-2Zr-4Al-1C............	42	31	x	-	-	y
11.....	Cb-15Hf-5W-2Zr-1C................	53	-	-	-	x	-
12.....	Cb-15Hf-2Zr-4V-1C................	53	-	-	-	x	-
13.....	Cb 10Ti-4Al-0.1Cu-2W.............	52	-	-	-	-	x
14.....	Cb-14.6Ti-3.9Zr-13.3Hf-3.5Al-0.7N	49	32	-	x	-	y
15.....	Cb-14.8Ti-3.9Zr-13.4Hf-3.4Si.....	53	36	x	-	-	y
Group 2:							
19.....	Cb-5Zr-5W........................	54	36	-	x	-	y
20.....	Cb-10Zr-5W.......................	55	38	-	x	y	-
21.....	Cb-15Zr-5W.......................	57	25	-	x	-	y
22.....	Cb-20Zr-5W.......................	51	23	-	x	-	y
23.....	Cb-33Zr-5W.......................	47	33	-	x	-	y
Group 3:[4]							
13a....	Cb-15Hf-5W.......................	51,54	-	-	-	x	x
24.....	Cb-15Hf-5W-0.1C..................	42,54	-	-	-	x	x
25.....	Cb-15Hf-5W-0.5C..................	49-55	-	-	-	x	x
26.....	Cb-15Hf-5W-1.0C..................	48-50	-	-	-	x	-
27.....	Cb-15Hf-5W-0.1N..................	49-51	-	-	-	x	x
28.....	Cb-15Hf-5W-0.5N..................	48-55	-	-	-	x	-
29.....	Cb-15Hf-5W-1.0N..................	52-55	-	-	-	x	-
30.....	Cb-15Hf-5W-0.1B..................	51-54	-	-	-	x	-
31.....	Cb-15Hf-5W-0.5B..................	54,55	-	-	-	x	x
32.....	Cb-15Hf-5W-1.0B..................	50,57	-	-	-	x	x
Group 4:							
16.....	Ta-5W-5Mo........................	48	-	-	-	-	x
17.....	Ta-15Hf-5W-2Re...................	44	24	x	-	y	-

[1]Poor--many cracks, no usable material available; fair--many cracks, but some usable material available; good--few edge cracks; excellent--no cracks.
[2]x--forging results at room temperature; y--forging results at 1,200° C.
[3]Failed at 1,500° C.
[4]Results are for duplicate alloys.

All alloys that could be forged were cleaned by etching in a solution of hydrofluoric and nitric acids and heat-treated for 5 hours in vacuum at 1,300° C. Previous experience with columbium alloys showed that recrystallization occurred with this combination of time and temperature. If the alloys were not sufficiently ductile to be cold- or hot-forged, no further tests were performed.

Rolling to a final thickness of 0.06 inch was done either at room temperature or at 1,000° C with 5 percent reductions each rolling pass. Samples for hot rolling were preheated 5 minutes. No protection from oxidation was provided.

The alloys in group 1 could not be rolled if silicon had been added. In group 2 only the alloys that were low in zirconium content were rolled successfully. All the alloys in group 3 were ductile to the extent that they could be cold rolled with only limited edge cracking. The tantalum-hafnium alloy containing both tungsten and rhenium was not sufficiently ductile to be rolled into sheet. The rolling data are shown in table 4.

TABLE 4. - Columbium and tantalum alloy rolling results

Alloy	Alloy composition, atomic percent	Rolling temperature	Reduction, percent	Results[1]
Group 1:				
1.....	Cb-1N-5W-3V-5Hf..................	Room temperature	67	Good.
2.....	Cb-1N-5W-5V-5Hf..................	.....do.........	71	Do.
3.....	Cb-1N-5W-3V-10Hf.................	.....do.........	70	Do.
4.....	Cb-1N-5W-5V-10Hf.................	.....do.........	61	Do.
5.....	Cb-1N-5W-3Si-5Hf.................	Room temperature and 1,000° C.	56	Fair.
6.....	Cb-1N-5W-3Cr-5Hf.................	Room temperature	63	Good.
10.....	Cb-15Hf-5W-2Zr-4Al-1C............	1,000° C........	59	Excellent.
11.....	Cb-15Hf-5W-2Zr-1C................	Room temperature	74	Good.
12.....	Cb-15Hf-2Zr-4V-1C................	.....do.........	65	Excellent.
13.....	Cb-10Ti-4Al-0.1Cu-2W.............	.....do.........	56	Do.
14.....	Cb-14.6Ti-3.9Zr-13.3Hf-3.5Al-0.7N	.....do.........	66	Good.
15.....	Cb-14.8Ti-3.9Zr-13.4Hf-3.4Si.....	.....do.........	69	Do.
Group 2:				
19.....	Cb-5Zr-5W........................	.....do.........	70	Excellent.
20.....	Cb-10Zr-5W.......................	1,000° C........	60	Do.
21.....	Cb-15Zr-5W.......................	Room temperature	76	Poor.
22.....	Cb-20Zr-5W.......................	1,000° C........	69	Center opened up.
Group 3:[2]				
13a....	Cb-15Hf-5W.......................	Room temperature	60,73	Good.
24.....	Cb-15Hf-5W-0.1C..................	.....do.........	67,68	Excellent.
25.....	Cb-15Hf-5W-0.5C..................	.....do.........	72,73	Do.
26.....	Cb-15Hf-5W-1.0C..................	.....do.........	70,71	Do.
27.....	Cb-15Hf-5W-0.1N..................	.....do.........	71,73	Do.
28.....	Cb-15Hf-5W-0.5N..................	.....do.........	67,69	Good.
29.....	Cb-15Hf-5W-1.0N..................	.....do.........	69,71	Do.
30.....	Cb-15Hf-5W-0.1B..................	.....do.........	66,72	Excellent.
31.....	Cb-15Hf-5W-0.5B..................	.....do.........	62,68	Do.
32.....	Cb-15Hf-5W-1.0B..................	.....do.........	63,67	Do.
Group 4:				
16.....	Ta-5W-5Mo........................	.....do.........	57	Do.
17.....	Ta-15Hf-5W-2Re...................	1,000° C........	53	Fair.

[1]Excellent--no cracks; good--few edge cracks; fair--many cracks but some usable material available; poor--many cracks, no usable material available.
[2]Results are for duplicate samples.

Strength

Elevated-temperature tensile tests were made at 1,200° and 1,400° C at a strain rate of 0.001 in/in/sec. The specimens were in the annealed condition and measured 0.04 inch thick, 0.250 inch wide, and 5 inches long, with no reduced gage section. Tests were made on a Marquardt[4] tensile testing machine described in a previous report (6). The alloys were electrically heated by self-resistance in a vacuum of 10^{-5} torr. A platinum versus platinum-10 percent rhodium thermocouple was spotwelded to the center of the specimen to sense the temperature. Heating time to temperature and holding time totaled about 1 minute.

Three of the columbium alloys tested at 1,200° C had tensile strengths near 40,000 psi (see table 5); alloy 4 of group 1 contained Cb-1N-5W-5V-10Hf, and alloys 31 and 32 of group 3 contained Cb-15Hf-5W, plus 0.5 and 1 atomic percent boron, respectively. In addition to solid-solution strengthening, precipitation hardening due to nitride or boride formation probably occurred. No exceptional values were observed for alloys in group 2 and 4. Since only one alloy composition in group 2 was fabricated into tensile specimens, no strength comparisons were made between this group and the Cb-15Hf-5W alloy.

Several low strength values were observed for some of the alloys tested; some showed defects such as inclusions, and some showed defects from fabrication. In several tests the samples failed outside the gage length. In most of these instances, the results were discarded. None of the current 13a (Cb-15Hf-5W) samples gave valid strength results; therefore results from a previous investigation (6) are reported in table 5.

Metallography

Microstructures of some of the alloys are shown in figure 1. They show that complete recrystallization has taken place and that one or more phases are distributed throughout the specimens. Alloy 13a (Cb-15Hf-5W) shows a solid-solution structure with a little second phase. The specimens used for metallography were cut from the cold portions of the tensile specimens. The alloys had been recrystallized at 1,300° C for 5 hours in a vacuum of 10^{-5} torr.

Oxidation Behavior

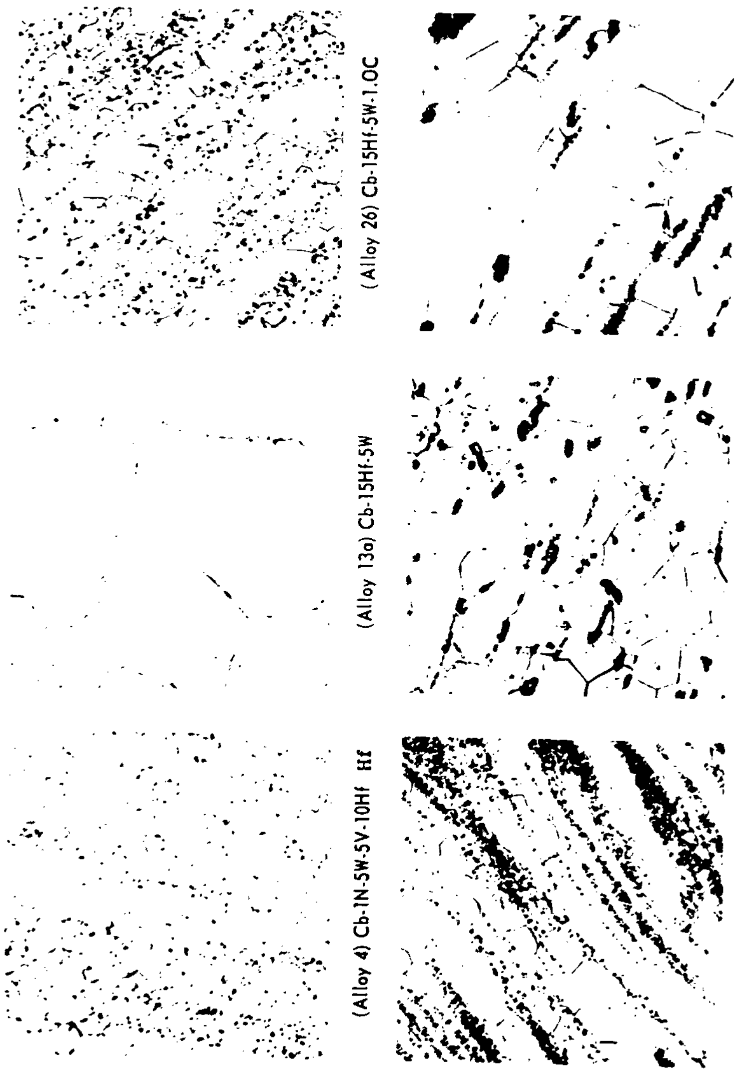
The alloys that rolled successfully were cut into samples measuring approximately 0.1 by 2 by 2 cm, cleaned, and recrystallized. To determine oxidation resistance, the specimens were tested in air for 2 hours at 1,000° and 1,200° C. Testing was done in a vertical tube furnace that was open on top and closed at the bottom. During oxidation testing, the weight of each sample was continuously recorded with an automatic recording balance.

[4] Reference to specific makes or models of equipment is made to facilitate understanding and does not imply endorsement by the Bureau of Mines.

TABLE 5. - Elevated-temperature tensile test data for columbium and tantalum alloys

Sample	Alloy composition	Testing temperature, ° C	Tensile strength 1,000 psi	Yield strength (0.2 percent offset) 1,000 psi	Elongation, percent in 1 inch	Type of fracture
1....	Cb-1N-5W-3V-5Hf..................	1,200	35.9	32.9	24	Chisel.
3....	Cb-1N-5W-3V-10Hf.................	1,200	33.0	33.0	34	Do.
4....	Cb-1N-5W-5V-10Hf.................	1,200	42.1	42.1	31	Do.
6....	Cb-1N-5W-3Cr-5Hf.................	1,200	35.0	30.1	15	Do.
6....	Cb-1N-5W-3Cr-5Hf.................	1,400	25.0	17.0	(1)	Do.
10....	Cb-15Hf-5W-2Zr-4Al-1C............	1,200	35.0	34.8	40	Do.
10....	Cb-15Hf-5W-2Zr-4Al-1C............	1,400	24.5	24.5	48	Do.
12....	Cb-15Hf-2Zr-4V-1C................	1,200	35.0	35.0	41	Do.
13....	Cb-10Ti-4Al-0.1Cu-2W.............	1,200	20.2	16.9	33	Do.
13....	Cb-10Ti-4Al-0.1Cu-2W.............	1,200	20.7	18.2	34	Do.
14....	Cb-14.6Ti-3.9Zr-13.3Hf-3.5Al-0.7N	1,200	21.4	18.2	63	Do.
19....	Cb-5Zr-5W........................	1,200	29.2	24.5	(1)	Brittle.
19....	Cb-5Zr-5W........................	1,400	20.9	20.0	36	Do.
24....	Cb-15Hf-5W-0.1C..................	1,200	36.9	32.2	41	Chisel.
24....	Cb-15Hf-5W-0.1C..................	1,200	33.8	28.4	12	Do.
25....	Cb-15Hf-5W-0.5C..................	1,200	35.9	31.8	18	Do.
25....	Cb-15Hf-5W-0.5C..................	1,200	30.6	29.6	(1)	Do.
25....	Cb-15Hf-5W-0.5C..................	1,200	35.1	32.4	37	Brittle.
26....	Cb-15Hf-5W-1C....................	1,200	35.3	32.1	12	Do.
30....	Cb-15Hf-5W-0.1B..................	1,200	37.8	30.8	6	Do.
31....	Cb-15Hf-5W-0.5B..................	1,200	45.0	(1)	4	Do.
32....	Cb-15Hf-5W-1B....................	1,200	38.3	(1)	(1)	Do.
32....	Cb-15Hf-5W-1B....................	1,200	39.4	33.6	(1)	Do.
32....	Cb-15Hf-5W-1B....................	1,200	40.0	36.4	25	Irregular.
13a[2]..	Cb-15Hf-5W.......................	1,200	42.0	37.6	26	-
13a[2]..	Cb-15Hf-5W.......................	1,315	35.2	34.6	7	-
13a[2]..	Cb-15Hf-5W.......................	1,480	27.4	25.5	41	-
16....	Ta-5W-5Mo........................	1,200	42.2	38.4	5	Irregular.
16....	Ta-5W-5Mo........................	1,400	33.4	30.9	8	Chisel.

[1] Fracture occurred outside gage length.
[2] Data from reference 6, p. 22, used.



(Alloy 4) Cb-1N-5W-5V-10Hf Hf

(Alloy 13a) Cb-15Hf-5W

(Alloy 26) Cb-15Hf-5W-1.0C

(Alloy 29) Cb-15Hf-5W-1N

(Alloy 31) Cb-15Hf-5W-0.5B

(Alloy 32) Cb-15Hf-5W-1.0B

FIGURE 1. - Selected Alloys Heat-Treated 5 Hours at 1,300° C in Vacuum and Furnaced-Cooled; Transverse of Tensile Sample (X 500).

As shown in figure 2, all the alloys in group 3 and alloy 4 (Cb-1N-5W-5V-10Hf in group 1) had low rates of oxidation. Alloy 4 also had high strength (42,000 psi) at 1,200° C. Over a 2-hour period the weight gain was 34 mg/cm^2 at 1,200° C and only 21 mg/cm^2 at 1,000° C. This is better than a twofold improvement over unalloyed columbium and about equal to alloys developed by other investigators (12). Oxidation resistance improved with increasing zirconium content in group 2 and compared favorably with the Cb-15Hf-5W alloy.

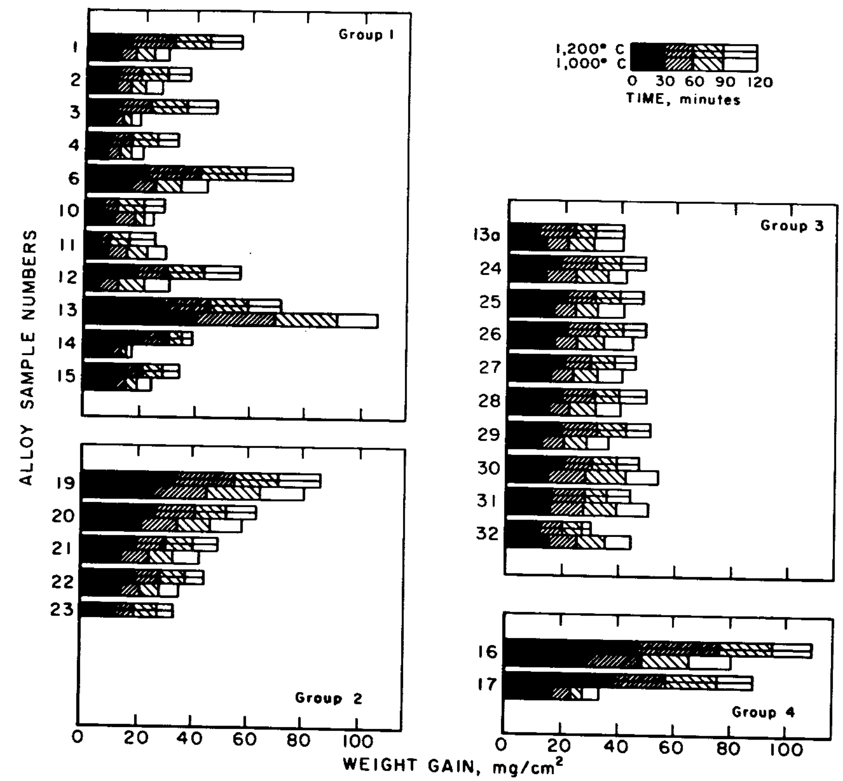


FIGURE 2. - Oxidation Data for Columbium and Tantalum Alloys.

CONCLUSIONS

1. The strength and oxidation resistance of columbium and tantalum may be increased substantially by combining with small amounts of other elements, while sufficient ductility may be retained to permit fabrication. Of the elements used for alloying, the most effective strengtheners were tungsten, vanadium, hafnium, nitrogen, and boron. Oxidation resistance was also improved.

2. Three alloys had tensile strengths of 40,000 psi at 1,200° C. The alloy compositions were as follows:

Cb-1N-5W-5V-10Hf (alloy 4)
Cb-15Hf-5W-0.5B (alloy 31)
Cb-15Hf-5W-1.0B (alloy 32)

3. The three strongest alloys also had good resistance to oxidation at 1,000° and 1,200° C. Better than a twofold improvement over unalloyed columbium was noted, and each alloy was about equal to some of the commercial alloys developed to date.

4. The three alloys listed have shown enough merit to warrant further investigation of their engineering properties. In addition a more intensified study is recommended for alloys containing boron additives to the base composition, Cb-15Hf-5W.

End

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